ATTACHMENT J

TO
BASIC ORDERING AGREEMENT
ATTACHMENT 1
STATEMENT OF WORK

MINIMUM
REQUIREMENTS
FOR
RADIOCHEMISTRY

PROCUREMENT OF ANALYTICAL SERVICES

MINIMUM REQUIREMENTS FOR RADIOCHEMISTRY

PROCUREMENT OF RADIOANALYTICAL SERVICES

MINIMUM REQUIREMENTS

PART 1

GENERAL RADIOANALYTICAL REQUIREMENTS

The purpose of this Statement of Work (SOW) is to provide a general standardized set of radioanalytical requirements that will ensure that high quality, consistent/comparable, and defensible analytical data is supplied by subcontracted and government labs that support the various DOE and DOE Contractor programs. Data quality objectives, which are project specific, are not addressed and may require modification to meet specific requirements.

1. RADIOANALYTICAL METHODS

This section contains general method requirements for analytical radiochemistry that are applicable to all radiochemical analysis. The requirements for specific analyses shall supersede any general requirements in the case of conflicting statements. General requirements applicable to all samples and not just to radiological samples are given in the *GENERAL REQUIREMENTS* of this Statement of Work.

1.1 SELECTION OF METHOD

Analytical methods selected to produce data to meet the requirements of this SOW shall not have conditions and limitations that can preclude the possibility of meeting the data requirements. This condition applies to sample preparation, separation, preparation for counting, and actual counting or measurement of the sample.

1.1.1 The analytical method selected shall be capable of producing data that meets the minimum method QA/QC requirements. All exceptions shall be approved by the Site prior to use.

1.1.2 Methods that can be referenced to nationally accepted sources such as EPA methods, DOE Methods Compendium, HASL 300 methods, etc. are preferred.

1.1.3 Methods selected and subsequent SOPs shall be tested and validated with control samples and blanks prior to analysis of Site samples.

1.2 SAMPLE HOLDING TIMES AND PRESERVATION REQUIREMENTS

1.2.1 **Holding Time**: The maximum sample holding time allowable under this contract is 180 days. In addition the maximum sample holding time shall not exceed five half-lives of an unsupported nuclide (see Glossary definition) of interest when five half-lives is shorter than 180 days. Sample specific guidance/requirements may be provided by the Site for specific isotopes or for very short-lived isotopes.

1.2.2 **Sample Preservation**:

1.2.2.1 The proper sample preservation will be the responsibility of the Site and will be indicated on the Chain of Custody (COC). It is the responsibility of the Site to indicate appropriate preservation on the Chain of Custody.

1.2.2.2 Samples other than water shall not be preserved with acid. For unusual matrices, sample specific guidance/requirements for verification of preservation may be provided on a case-by-case basis.

1.2.2.3 The laboratory shall verify and document preservation of samples upon receipt.

1.2.2.4 If samples, which are not preserved as indicated on the COC, are received, the laboratory shall contact the Site immediately.

1.2.2.5 The laboratory shall not perform any pH adjustment of Site samples unless prior approval is obtained from the Site.

1.2.2.6 All issues associated with samples, which are not preserved as indicated on the COC (and their resolution), shall be reported in the Case Narrative.

1.3 RETENTION OF UNUSED SAMPLE(S)

The laboratory shall retain unused samples, dissolved samples, and counting preparations as specified in Section 3.1.3.4 of the *General Requirements* of this SOW.

1.4 RADIOLOGICAL CONTROL

The laboratory shall maintain a radiological control program that rigorously addresses analytical radiological control. The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low level and high level samples will be identified, segregated and processed in order to prevent sample cross-contamination.

1.5 WATER PURITY

Water purity shall be at least distilled or deionized water, or as specified on a case-by-case basis by the Site. Further water purity guidance/requirements are found in the analysis specific requirements of this SOW.

1.6 PREPARATION REQUIREMENTS FOR SOLID SAMPLES

1.6.1 Due to the diverse nature of samples, comprehensive specification for all sample preparation for subsampling is not practicable. In all cases, the contract laboratory shall make every effort to assure that any aliquots or subsamples for analysis are representative of the bulk sample. Sample specific guidance/requirements may be provided by the Site on a case-by-case basis.

1.6.2 When solid samples are dried, the results shall be reported on a dry basis and the percent moisture shall be reported unless otherwise specified by the Site.

1.7 PREPARATION REQUIREMENTS FOR SAMPLES CONTAINING SUSPENDED SOLIDS

1.7.1 Sample specific guidance/requirements for analysis of the total sample or separate analysis of solution and suspended solids will be provided by the Site either prior to sample receipt or within the sample documentation accompanying the samples.

1.7.2 If filtration is required, sample specific guidance/requirements will be provided regarding filter type and mesh size requirements by the Site either prior to sample receipt or as part of the sample documentation accompanying the samples.

2. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

This SOW requires a variety of activities that represent the minimum quality assurance/quality control (QA/QC) operations necessary to satisfy analytical quality requirements. These operations and those in the *General Requirements* of this SOW are designed to ensure that data produced meets defined minimum

data quality objectives. These requirements do not release the Laboratory from maintaining its own QC checks on method and instrument performance.

2.1 QUALITY ASSURANCE PLAN

Requirements for the quality assurance plan are specified in the *General Requirements* of this SOW.

2.2 ANALYTICAL BATCH QUALITY CONTROL REQUIREMENTS

A batch of samples is 20 samples or less plus a batch blank, a laboratory control sample, a duplicate and a matrix spike as appropriate. A batch of samples is processed throughout the entire analytical process together. If equipment restrictions limit the number of samples in any particular step, the samples in the batch shall be processed continuously and consecutively until the entire batch is completed.

2.2.1 Sample Control: All laboratory identifications(ID) used for the prepared samples through the entire analysis (e.g. identifications of beakers, planchets, filter papers, vials, or their holders, etc.) shall be documented and traceable to the Site sample identifications and the respective preparation Analytical Batch Identification.

2.2.1.1 The Analytical Batch ID for customer requested reanalysis shall be different from that of the original analysis. The exact form of the identification may be defined by the laboratory as to whether the ID shall be completely different or may have the original ID with an extension. All reanalysis shall be traceable to the reanalysis Analytical Batch Identification and to the reanalysis Analytical Batch QC samples.

2.2.2 QC Sample Identification: All QC samples in the Analytical Batch shall be identifiable as to QC type (e.g., Duplicate, Blank, Laboratory Control Sample[LCS], Matrix Spike) and shall be traceable to the preparation Analytical Batch identification.

2.2.3 QC Material Traceability

2.2.3.1 All tracer, carrier, matrix spike, and LCS aliquots shall be traceable to their respective primary standard reference material (SRM) certificate (if applicable, minimally to the reagent lot number and supplier, the standard log, and the respective preparation Analytical Batch ID).

2.2.3.2 All QC materials shall have identified expiration dates (lab assigned for all preparations and any stock materials with no expiration dates provided by the supplier). No QC materials shall be used beyond their expiration dates. Results associated with expired quality control materials are not valid.

2.2.3.3 Tracer, LCS, and matrix spike materials shall be prepared from the National Institute of Standards and Technology (NIST) traceable standards. If a NIST traceable standard reference material cannot be procured, then the standard shall meet the requirements for a "working reference material" as described in STD.ASTM C1128.

2.2.4 Traceability of Measuring and Testing Equipment (M&TE): All pipet and balance identifications specified on all Analytical Batch preparation benchsheets or logs shall be traceable to the respective calibration log.

2.2.5 QC Sample Preparation: All samples and QC samples in each Analytical Batch shall be prepared concurrently and in the same manner.

2.2.6 QC Sample Counting: All QC samples shall be counted and analyzed in the same manner as the samples in the Analytical Batch, in the same time frame and using the same instrument calibration parameters, instrument analysis algorithms, etc.

2.2.6.1 The same time frame implies that where multiple detectors are used and are sufficient to count the entire batch at the same time, the entire batch is counted at the same time. If the number of detectors is not sufficient to count the entire batch at the same time, then samples shall be counted consecutively on the available detector(s).

2.2.6.2 The same instrument calibration parameters, instrument analysis algorithms, etc. implies that these parameters for a given instrument shall not be changed for the samples in that batch. It is understood that for multiple detectors, the parameters may not be identical.

2.3 BATCH QUALITY CONTROL SAMPLES

2.3.1 Batch Blank The batch blank is a laboratory-generated sample prepared with absence of the analyte of interest. Batch blanks are batch quality indicators and are carried through the entire sample analysis procedure with the samples in the batch.

2.3.1.1 Matrix: Sample specific guidance will be provided by the Site either prior to sample receipt or as part of the sample documentation accompanying the samples on a case-by-case basis for the batch blank matrix. If no guidance is provided the matrix shall be the same as the samples, as can be reasonably achieved and shall be documented in the Case Narrative.

2.3.1.2 Frequency: At least one batch blank shall be prepared and analyzed with every Analytical Batch of samples.

2.3.1.3 Counting: Batch blanks shall be counted for a sufficient time to meet the required detection limit except in the case where the achieved MDA is calculated from the standard deviation of a blank population. In this case the batch blanks shall be counted for the same count time as the samples.

2.3.1.4 Acceptance Criteria:

- The MDA of the batch blank shall be less than the RDL unless all samples in batch are positive, as defined by the Site.
- If all sample results in the batch are greater than the RDL, then the Batch Blank MDA shall be less than the activity of the least active sample in the batch of that sample.
- If all of the samples in the batch are less than the RDL, the activity of the blank shall be less than the MDA.
- Refer to Section 2.7 for reanalysis requirements.

2.3.1 Laboratory Control Sample (LCS) The laboratory control sample is a quality indicator and provides information about the relative bias of the analysis. It is used to assess the overall process for any inherent biases or trends. The LCS contains known quantities of analyte and is carried through the entire analysis procedure with the samples.

2.3.2.1 Frequency: At least one LCS shall be prepared and analyzed with every Analytical Batch of samples.

2.3.2.2 Selection and Level: The LCS shall be of the same analyte as the sample analyte and shall be at least 5 times but not greater than 20 times the RDL with the following exceptions:

- For RDLs of low activity the analyte shall be at a level where the random counting error does not exceed 10% in the counting time required to attain the RDL.
- Analytes for gamma spectroscopy need not be the same as the sample analyte but should fall in the approximate energy region of the spectrum (low, mid-range, or high energy).
- For gross alpha, gross beta analysis, Site specific guidance will be provided for selection of the analyte and acceptable relative bias when the analyte differs from that used for the calibration curve.

2.3.2.3 Counting: The LCS shall be counted for a sufficient time to meet the required detection limit.

2.3.2.4 Matrix: Sample specific guidance will be provided by the Site either prior to sample receipt or as part of the sample documentation accompanying the samples on a case-by-case basis for the LCS matrix. If no guidance is provided the matrix shall be the same as the samples, as can be reasonably achieved, and the matrix shall be documented in the Case Narrative.

2.3.2.5 Acceptance Criteria:

The relative bias as calculated from the formula:

$$\textbf{Relative bias} = \frac{observed - known}{known}$$

shall be in the range -.25 to +.25. (Reference: ANSI N13.30, Appendix B)

- For gross alpha, gross beta analysis, the acceptance criteria are applicable when the analyte in the LCS is the same analyte used for the calibration curve. Site specific instructions will be provided when the LCS analyte is different from that used for the calibration curve.
- Site/Project specific requirements may be provided.
- Refer to Section 2.7 for reanalysis requirements.

2.3.3 Duplicates The purpose of the Duplicate sample analysis is to assess the Laboratory precision by providing information on the Laboratory's reproducibility, and the homogeneity of the sample. The Duplicate activity shall <u>not</u> be averaged with the corresponding sample activity when reporting results. Samples identified as Field Blanks shall not be used for Duplicate sample analysis. The Site may require that a specific sample be used for Duplicate sample analysis.

2.3.3.1 Frequency: At least one duplicate sample shall be prepared and analyzed with every Analytical Batch of samples.

2.3.3.2 Counting: The duplicate shall be counted for a sufficient time to meet the required detection limit.

2.3.3. 3 Evaluation Criteria: The normalized absolute difference between the sample and laboratory duplicate, given by the following equation shall be used to

determine that the results do not differ significantly when compared to their respective one sigma uncertainty.

$$\frac{S - D}{\sqrt{(TPU_S)^2 + (TPU_D)^2}} \leq 3^*$$

- (* 2.58 was rounded to 3)
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- S = Sample result
- D = Duplicate result
- TPU_S = 1s Total Propagated Uncertainty of the sample
- TPU_D = 1s Total Propagated Uncertainty of the duplicate

- Duplicates, which do not meet the above requirements due to the difficulty of subsampling, shall be described in the Case Narrative.

2.3.4 MATRIX SPIKES: Matrix spikes consist of analysis of a replicate of an actual sample to which a known quantity of the analyte has been added. Recovery (determined as the percentage of "found" analyte relative to the known amount introduced) provides information on sample specific matrix effects that result in an analytical bias for a given analysis batch. (e.g. H-3, C-14, etc.) Matrix Spikes shall be added as early in the sample preparation steps as practicable.

- Matrix spikes are not required for radiochemical analyses if an isotopic tracer or chemical carrier is used in the analysis to determine chemical recovery (yield) for the chemical separation and sample mounting procedures. Matrix spikes are not required for Gross Alpha, Gross Beta, or Gamma Analysis.
- Matrix spikes shall be run on a separate sample aliquot using the same analyte as that being analyzed whenever possible.

2.3.4.1 Frequency: The matrix spike shall be prepared and analyzed at the frequency of one matrix spike per Analytical Batch. A batch is 20 or fewer samples.

2.3.4.2 Selection and Level: The matrix spike shall be added at a concentration of at least 5 but not greater than 20 times the RDL. In samples having known significant activity of the radionuclides to be analyzed, more than 20 times the RDL may be added to minimize the effect of the sample activity on determination of spike recoveries.

2.3.4.3 Counting: The matrix spike shall be counted for a sufficient time to meet the required detection limit.

2.3.4.4 Acceptance Criteria: Matrix spike recoveries shall be within the control limits of 60 – 140%. Matrix spike samples for which the sample activity is greater than five times the spiking level are not required to meet this criteria.

2.3.4.5 Refer to section 2.7 for reanalysis requirements.

2.4 RECOVERY OF TRACERS AND STABLE CARRIERS

Isotopic tracers are typically radioactive materials (e.g., Pu-242, Sr-85) while carriers are typically nonradioactive (e.g., natural strontium). They are added to samples to determine the overall chemical yield for the analytical preparation steps. When tracers or carriers are used, each sample (including any batch associated QC samples) shall be "spiked" separately with the same materials and individual sample yields will be determined. The tracer shall be added to the

sample at the very beginning of the sample preparation procedure. For solid samples the tracer shall be added after grinding, sieving, etc. but prior to any muffling or dissolution of the sample.

2.4.1 Isotopic Tracers: The recovery of isotopic tracers shall be in the range 30% - 110%.

2.4.2 Stable Carriers: The recovery of stable carriers shall be in the range 40% - 110%.

2.4.3 Refer to Section 2.7 for reanalysis requirements.

2.5 RESULTS REPORTING REQUIREMENTS

2.5.1 Reporting Figures: All reported quantities including but not limited to result, total propagated uncertainty, and minimum detectable activity shall be reported to three digits in scientific notation.

2.5.2 Negative Numbers: All negative activities shall be reported as such. If the sum of the activity and the measurement uncertainty at 3-sigma is a negative number, the cause shall be investigated and evaluated to determine if it is systematic or random. If the cause is systematic, it shall be corrected. If the cause is random it shall be documented in the Case Narrative. Recurrent problems with significant negative results suggest that the background subtraction and/or blank subtraction, if applicable, are in error or that the estimate of error is low. Investigation of such problems and documentation of the resolution is required and shall be discussed in the Case Narrative.

2.5.3 Total Propagated Uncertainty: All measurement uncertainties shall be propagated and reported with each result. The formula for calculating the total propagated uncertainty of a result shall be documented in the appropriate SOP. The total propagated uncertainty shall include both systematic and random error.

2.5.3.1 Systematic Error shall include but is not necessarily limited to:

- the errors from all measurement devices such as but not limited to pipets and balances.
- the uncertainty of known values of tracer solutions, calibration uncertainties, etc.

2.5.3.2 Random Error shall include but is not necessarily limited to the total random counting error associated with each sample and appropriately propagated when more than one variable is used to determine the result.

2.6 MINIMUM DETECTABLE ACTIVITY (MDA) DETERMINATION

2.6.1 The MDA (mimimum detectable amount) is the smallest amount (activity or mass) of an analyte in a sample that will be detected with a probability β of non-detection (Type II error) while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). For the purposes of this SOW, the α and β probabilities are both set at 0.05 unless otherwise specified. (Reference: *ANSI N13.30 and ANSI N42.23*)

2.6.2 MDA Factors and Conditions: MDAs are determined based on the factors and conditions, which influence the measurement. Sample size, count duration, tracer

chemical recovery, detector background, blank standard deviation, and detector efficiency shall be optimized to result in sample MDAs less than or equal to the RDLs. If RDLs are not achieved, then the cause shall be addressed comprehensively in the Case Narrative.

2.6.3 MDA Calculation: The basic detection limit calculation shall be based on that developed by L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination, ANALYTICAL CHEMISTRY, March, 1968, Vol. 40, NO.3, pg. 586.The following general equations shall be used to calculate the MDA. Sample specific requirements may be provided on a case by case basis.

- **WITHOUT BLANK POPULATION**:

$$MDA = \frac{4.65 * \sqrt{\frac{b}{T}}}{K} + \frac{3}{K * T}$$

where,

b = background count rate in cpm

T = count time in minutes

K = efficiency * $e^{-\lambda t}$ * aliquot fraction * tracer recovery*ABN

Efficiency = detector efficiency

t = time from sample collection to mid-point of count time(or nuclide separation time, as applicable) in the same units as half-life

λ = Analyte decay constant = ln2/(half-life)

ABN = abundance

- ❖ Use of the above equation requires that the background and sample count times are either equivalent, or the background count time is greater than the sample count time. When sample and background counts are different, this must be included in the equation.
- ❖ The above equation for MDA has the units of dpm/sample. Any other units will require specification by Site/Project.
- ❖ Site specific requirements may be provided for other MDA formulations.

- **WITH BLANK POPULATION**

$$MDA = \frac{4.65 * s_b}{KT} + \frac{3}{KT}$$

The use of this equation assumes that the standard deviation of a sample, where the sample contains no actual analyte activity above that of the appropriate blank, is equal to the standard deviation of the appropriate blank. For any other implementation, Site specific instructions will be provided.

s_b = standard deviation of the blank population where the blank population is in total blank counts in count time T

Use of blank populations for calculation of MDAs requires the selection of an implementation method, which includes but is not limited to:

- Identification of blanks to be used in the population
- The number of blanks to use in the population
- How the blank population changes
- Limitations on the deletion of blanks
- The method of implementation shall not introduce any statistical bias
- The appropriate blank subtraction shall be the mean blank value of the blank population

  ❖ The implementation of blank populations for calculation of MDAs shall be described in detail in an SOP, which is accepted by the Site prior to use.

(Reference: *ANSI N13.30,* Appendix A.5.13 for examples of MDA calculations)

2.6.4 MDA Optimization: The laboratory shall optimize analysis parameters in order to achieve analyte MDAs less than or equal to the RDLs, except when sample activities are significantly greater than the RDL. Samples with elevated activities shall be handled according to the following requirements:

2.6.4.1 The appropriate aliquot size shall be determined based on the activity level in the sample. The aliquot shall be large enough to generate data, which meet the following criteria:

2.6.4.2 The measurement uncertainty shall not be greater than 10% of the sample activity.

2.6.4.3 The MDA for the analysis shall be a maximum of 10% of the sample activity.

2.7 CONDITIONS REQUIRING REANALYSIS

If reanalysis is not possible, the Site shall be contacted for specific gudiance/requirements.

2.7.1 General Conditions

2.7.1.1 If the RDLs could not be achieved because of laboratory errors or oversights such as inadequate count times, inadequate aliquot size, inappropriate dilution, low detector efficiencies, high detector backgrounds, etc., then the sample shall be reanalyzed under more optimal conditions.

2.7.1.2 If the RDLs could not be achieved because of problems associated with the sample such as inadequate sample provided, elevated radioactivity levels, sample matrix interferences such as high amounts of suspended solids, multiphase liquids, etc., then such problems shall be explained in the Case Narrative.

2.7.2 Sample and Analyte Specific Conditions: Any one of the following are additional conditions that require <u>reanalysis for a particular sample and analyte:</u>

2.7.2.1 If, for any reason, sample or batch QC integrity becomes suspect (e.g., spillage, mis-identification, cross-contamination), all potentially affected samples shall be reanalyzed from a point before that which the integrity came into question. If new batch QC must be prepared for reanalysis, samples for reanalysis shall be restarted at the normal point of initiation for the batch QC.

2.7.2.2 All samples failing the criteria for tracers or carriers as defined in Section 2.4.
2.7.2.3 All samples associated with expired standards.

2.7.3 Analytical Batch Conditions: Exceptwhere noted otherwise, any one of the following conditions requires reanalysis of the entire Analytical Batch, beginning with the preparation:

2.7.3.1 Batches, which failed the Batch Blank criteria for, batch blanks as defined in Section 2.3.1.
2.7.3.2 Batches, which failed the LCS criteria as, defined in Section 2.3.2.
2.7.3.3 Batches, which failed the Matrix Spike criteria as, defined in Section 2.3.4.

2.8 CONDITIONS REQUIRING A RE-COUNT:

2.8.1 If the RDL was not achieved due to inadequate count duration, low detector efficiencies, or high detector backgrounds, the sample shall be re-counted under more optimal conditions, and the reasons for the re-count shall be documented in the Case Narrative.
2.8.2 Additional Site specific requirements may be provided for re-counts.

2.9 VERIFICATION OF STANDARDS PREPARTIONS: In addition to the general requirements pertaining to all standards as defined in the *General Requirements* of this SOW, the following requirements for verification of prepared standards shall be observed.

2.9.1 Standards shall be verified prior to initial use.
2.9.2 Preparations of standards solutions used for a period of time exceeding one year shall be verified annually, at a minimum, and documented in a logbook.
2.9.3 At least three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.
2.9.4 The certificate value (NOT including any uncertainty) shall lie within the 95% confidence interval determined from the mean and two sigma standard deviation of the three measurements.
2.9.5 The two sigma value used for the 95% confidence interval shall not exceed 10% of the mean value of the three verification measurements.

2.10 MEASURING AND TESTING EQUIPMENT REQUIREMENTS

Requirements for measuring and testing equipment are specified in the *General Requirements* of this SOW.

2.11 DATA MANAGEMENT

Requirements for Site data management are specified in the *General Requirements* of this SOW or in Site specific SOWs.

2.12 PERFORMANCE EVALUATION (PE) SAMPLES

Participation in the following evaluation programs is required under this statement of work. Performance Evaluation Samples are discussed further in the *General Requirements* of the SOW.

- DOE Quality Assessment Program
- DOE Mixed Analyte Performance Evaluation Program (MAPEP)

2.12.1 Level of Participation: The Laboratory shall participate in an interlaboratory comparison study for each method used for this SOW and for which DOE QAP and MAPEP provide performance evaluation samples and for which they perform analyses.
2.12.2 Frequency: Semi-annually

2.12.3 Unacceptable Performance: Reporting an unacceptable value falling outside the warning limits, as calculated by the program, will result in a probationary period until the next reporting period for that analyte. If the Laboratory fails two consecutive evaluations, the Laboratory will not receive samples for analysis by the failed method until an acceptable PE score has been achieved or other verification of corrective action. Root cause and corrective action reports for PE samples outside of acceptable limits are to be submitted to the potentially affected Site(s) within 21 days from receipt of the scores.

2.12.4 PE Score Disclosure: PE scores and names of laboratories under this SOW will be available to the DOE and any other appropriate organization and/or individual procuring analytical services for DOE.

2.13 INSTRUMENT MAINTENANCE/REPAIR DOCUMENTATION

2.13.1 The laboratory shall identify and document the instrument manufacturer, model number, configuration, settings, detector identifications, and any modifications in the instrument maintenance log.

2.13.2 All repairs and modifications shall be documented.

2.13.3 Following all repairs and modifications, verification of calibration and background determination and/or calibration with background determination shall be performed and documented.

2.14 ON-SITE LABORATORY EVALUATIONS

Requirements for on-Site laboratory evaluations are specified in the *General Requirements* this SOW.

2.15 PERFORMANCE CRITERIA

Requirements for performance criteria are specified in the *General Requirements* this SOW.

PART 2

ISOTOPIC DETERMINATIONS BY ALPHA SPECTROMETRY

1. RADIOCHEMICAL ANALYTICAL METHODS

1.1 INTERNAL TRACER METHOD

1.1.1 The internal tracer method shall be used for isotope specific analysis by alpha spectrometry.

1.1.2 The tracer shall be added to the sample at the very beginning of the sample preparation procedure. For solid samples tracer shall be added after grinding, sieving, etc. but prior to any muffling or dissolution of the sample.

1.1.3 Initial sample preparation shall include treatment to ensure that tracer and analyte will undergo similar reactions during processing.

1.1.4 All tracers used for alpha spectrometry shall be tested by the lab for contribution in the ROIs of the analytes of interest. If a significant contribution is found, the method for correction shall be Site accepted prior to use.

1.2 BACKGROUND CORRECTION

1.2.1 The gross counts in each target analyte and tracer region of interest (ROI) shall be corrected for the particular detector's background contribution in those same ROIs.

1.2.2 Site/Project specific instructions may be provided regarding the required background subtraction.

1.3 BLANK CORRECTION

1.3.1 Blank correction shall not be routinely performed.

1.3.2 When blank correction is necessary, requirements will be established as Site or Project specific requirements. All blank correction requirements will be communicated to the laboratory prior to award of contract or contract modification.

2. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

2.1 CONDITIONS REQUIRING REANALYSIS

2.1.1 **Sample and Analyte Specific Conditions** Any one of the following are additional conditions that require reanalysis for a particular sample and analyte.

2.1.1.1 If the tracer recovery for the sample does not fall within 30% - 110%, reanalysis is required, beginning with preparation.

2.1.1.2 If the full width half maximum (FWHM) for the tracer peak exceeds 100 keV and/or the peak energy does not fall within ±50 keV of the known peak energy, reanalysis is required.

2.1.1.3 If the target analyte and tracer peaks are not resolved because the target analyte activity is significantly larger than the tracer activity, the sample shall be re-analyzed with a smaller aliquot such that resolution of tracer and analyte peaks is accomplished. Site specific guidance may be provided on a case by case basis for adequate resolution of tracer and target analyte.

2.1.1.4 If the sample analyte spectrum contains significant interferences with the analyte and/or tracer ROIs, reanalysis is required.

2.1.2 Analytical Batch Conditions Except where noted otherwise, any one of the following conditions requires reanalysis of the entire Analytical Batch, beginning with the preparation:

2.1.2.1 If the tracer chemical recovery for the Batch Blank does not fall with 30% - 110%, reanalysis is required if sufficient sample is available.

2.2 INSTRUMENT CALIBRATION

The purpose of the instrument calibration is to ensure that alpha spectrometry detectors used for sample analysis are capable of producing quality results according to the specifications given in this section, and that the calibration was maintained throughout the time period in which samples were analyzed.

2.2.1 Calibration: Calibration of each alpha spectrometry detector used to produce data for this SOW shall include:

- channel vs. energy calibration,
- efficiency determination
- and background determination for each ROI.

2.2.2 Frequency of Calibration

2.2.2.1 Channel vs. energy calibration shall be done at least monthly.

2.2.2.2 Background determinations for each ROI shall be done at least monthly.

2.2.2.3 Actual efficiency determinations shall be performed when the check source count is outside of the acceptable limits of the control chart (Reference: *ANSI N42.23*, Annex A5). Check source counts shall be done at least monthly.

2.2.2.4 Calibration for energy and background determination and efficiency determination shall be performed when a new detector is put into service or if repair is performed on an existing detector.

2.2.2.5 Site specific requirements may be provided when more frequent calibration is required.

2.2.3 Calibration Standards

2.2.3.1 Efficiency determinations shall be performed with sources, which are themselves NIST traceable or with sources prepared from NIST traceable standards.

2.2.3.2 When sources used for determinations for efficiency are prepared from NIST traceable standards, they shall be "working reference materials" as defined in *STD.ASTM C1128*. A material balance check shall be done on each source which clearly demonstrates that greater than 99% of the standards used were carried on the source. The material balance check shall be done on the fraction remaining from either the neodymium fluoride precipitation or the electrodeposition plus all rinses from an adequate cleaning of any vessel used in

the process. The estimated error in preparing the source shall be propagated into the error of the efficiency determination.

2.2.3.3 Check sources shall be used only to verify that efficiencies have not changed. They shall not be used to determine efficiencies.

2.2.4 Energy Calibration Requirements

2.2.4.1 The energy calibration for each detector shall be performed. A curve shall be fit for Energy (Y-axis) versus Channel (X-axis), and the equation with the slope and Y-intercept for the fit shall be documented.

2.2.4.2 The slope of the equation shall be $\leq$15 keV/channel.

2.2.4.3 The energy calibration shall be performed using at least three isotopes within the energy range of 3 to 6 MeV.

2.2.4.4 The final peak energy positions of all observed isotopes shall be within ±40 keV of the expected peak energy.

2.2.5 Background Requirements

2.2.5.1 The Background total counts (or counts per unit time) for each target analyte and tracer isotope ROI shall be analyzed on each detector and documented.

2.2.5.2 The Background for each ROI shall be sufficiently low to optimize the MDA.

2.2.5.3 The limits of acceptability for each background ROI shall be documented. These shall be set such that RDLs can be obtained for backgrounds at the limit of acceptability.

2.2.5.4 Background count times shall be equal to or longer than sample count times.

2.2.6 Efficiency Determination Requirements

Detector efficiency is not used in the calculation of results when tracers are used in the analysis, but only used to calculate the estimated yield, which is also not used, except as a general method performance indicator.

2.2.6.1 The Efficiency counts for the ROI shall be background corrected using the same ROI for the background unless the background is less than 0.5% of the total counts in the ROI.

2.2.6.2 The Efficiency shall be determined on at least 10,000 net counts in the ROI (after background correction).

2.2.6.3 Check source counts to verify efficiency shall be determined on at least 2,000 counts.

2.2.6.4 The Efficiency and Efficiency error shall be documented.

2.2.6.5 The efficiency check as determined by the check source count and its associated error and limits of acceptability for the check source result shall be documented.

2.3 SPECTRUM ASSESSMENT

2.3.1 ROIs shall be clearly indicated either graphically or in tabular form on alpha printouts.

2.3.2 The FWHM resolution for each sample and QC sample tracer peak shall be $\leq$100 keV.

2.3.3 The tracer peak energy for each sample and QC sample shall be within ±50 keV of the expected.

2.3.4 Each sample and QC sample spectrum shall be assessed for correctly chosen ROIs, acceptable spectral resolution, acceptable energy calibration and interferences with the analyte and tracer ROIs.

PART 3

LIQUID SCINTILLATION COUNTING

1. RADIOCHEMICAL ANALYTICAL METHODS

1.1 SAMPLE PREPARATION

1.1.1 **Tritium in Water**: Unless Site specific requirements are provided, water samples for tritium analysis and all associated QC samples shall be distilled prior to analysis. The applicable preparation SOP shall specify the fraction to be collected. The same fraction shall be collected for samples and all associated QC samples.

1.1.2 **Other**: (e.g. C-14, Fe-55, Ni-63, Tc-99, I-129, I-131, Pm-147, Pb-210, Pu-241) Due to the variety of effective methods for the analysis of various matrices and analytes, specific sample preparation procedures shall be accepted by the Site prior to use.

1.2 COUNTING VIAL PREPARATION

1.2.1 Samples shall be counted in vials equivalent to or superior to low potassium glass vials or high density polyethylene vials. Samples in polyethylene vials shall be counted within a time period not to exceed the manufacturer's specification for the cocktail used in the analysis. Analysis documentation shall contain sufficient information for this to be verified.

1.2.2 Vials shall be prepared according to manufacturer's specification for the cocktail. The vials shall be "dark adapted" for a minimum of 30 minutes or according to the cocktail manufacturer's specifications before counting. The prepared vials shall be inspected to verify that the sample loaded properly in the cocktail.

2. COUNTING AND CALCULATION REQUIREMENTS

2.1 Procedures for methods using liquid scintillation counting shall incorporate and adhere to ANSI N42.15-1997, *American National Standard Check Sources for and Verification of Liquid Scintillation Systems*. References are for the customer determined current version. When references change, an implementation schedule will be determined.

2.2 **Instrument Set-Up:** The instrument shall be set up according to the manufacturer's instructions. Any deviations shall be documented in the instrument maintenance log.

2.3 **Instrument Background:**The instrument background vial for all tritium matrices shall be prepared with low-tritium or "dead" water. The instrument background vial shall be prepared with the same water to cocktail ratio as the samples are prepared. The type of water used to prepare the instrument background vial shall be explicitly noted on the preparation and counting documentation. For other matrices and analytes, refer to the Site specific accepted procedure. The instrument background shall be determined weekly or with each sample batch. Unless calculated from a running average of background counts, the most recent background count shall be used to calculate sample activities and MDAs.

2.4 Efficiency: Instrument performance shall be checked each day of use.

2.4.1 For analysis methods using quench curves to determine individual sample counting efficiency, the quench curves shall be generated at least yearly and verified after any instrument maintenance.

2.4.2 If the calibration method is constant quench, the efficiency standards shall be counted weekly or with each counting batch.

2.4.3 Other methods for determining efficiency, such as internal-standard methods, shall be performed according to Site accepted procedures.

3. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS (Refer to the General Radiochemical Requirements)

3.1 Sample Specific Conditions In addition to the general requirements, the following are conditions that require reanalysis for a particular sample and analyte, beginning with the preparation or recounting, as appropriate:

3.1.1 If constant quench method of calibration is used, the quench of the sample shall be within +/-5% of the quench of the efficiency standard. If this condition is not met, the sample must be reanalyzed beginning with vial preparation.

3.1.2 If the sample quench does not fall within the range of the quench curve, the samples shall be re-analyzed such that the sample quench is in the range of a quench curve.

3.1.3 Site specific guidance may be provided for other methods such as counting “sample” and “sample plus spike”.

3.2 Spectrum Assessment: For analytes requiring separations other than distillation:

3.2.1 Sample spectra shall be retained for each sample and QC sample including identification of ROIs

3.2.2 Each sample and QC sample spectrum shall be assessed for correctly chosen ROIs, acceptability of peak shape, and interferences due to non-target analytes or luminescence.

PART 4

GAS FLOW PROPORTIONAL COUNTING

1. RADIOANALYTICAL METHODS

1.1 PLANCHET PREPARATION

1.1.1 Planchets shall be thoroughly cleaned before use to ensure that there are no interfering residues or contamination.

1.1.2 All planchets shall be prepared not to exceed sample weights in excess of the calibrated ranges of established self-absorption curves.

1.1.3 Sample weights shall be documented and stable prior to counting.

1.1.4 Planchets exhibiting physical characteristics notably different from the self-absorption standards (e.g., evidence of corrosion) shall not be counted unless remediation efforts such as additional sample preparation and remounting, flaming prove unsuccessful.

1.1.5 Any non-routine counting situations shall be documented in the Case Narrative.

2. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

2.1 LABORATORY CONTROL SAMPLE (LCS)

2.1.1 **Isotopes**: The isotopes used for the LCS for gross alpha/gross beta analysis shall be documented in the Case Narrative. Site specific guidance/requirements may be provided for isotopes used for the LCS.

2.1.2 **Evaluation Criteria:** For gross alpha/beta analysis, the acceptance criteria given in *Part 1, General Radioanalytical Requirements* of this SOW is applicable when the analyte in the LCS is the same analyte used for the calibration curve. Site specific instructions will be provided when the LCS analyte is different from that used for the calibration curve.

2.2 INSTRUMENT CALIBRATION

2.2.1 **Instrument** calibration shall be done in accordance with the requirements in ANSI N42.25, *Calibration and Usage of Alpha/Beta Proportional Counters.* Where the word "should" is used in ANSI N42.25, calibration shall be performed in accordance with the statement unless Site accepted justification is provided. References are for the customer determined current version. When references change, an implementation schedule will be determined.

2.2.2 Calibration Sources and Standards:

2.2.2.1 The standard reference material used to prepare sources for determining detector efficiencies and self-absorption curves shall be NIST traceable.

2.2.2.2 The calibration sources shall provide adequate counting statistics over the period for which the source is to be counted. However, the source shall not be so radioactive as to cause pulse pileups or dead time that is significantly different from that to be expected from routine analyses.

2.2.2.3 The geometry of the calibration sources used for efficiency and self-absorption/crosstalk curves shall be the same as that of the prepared sample and QC sample planchets. The depth and shape (flat, flanged, ringed, etc.), in addition to the diameter, are factors which shall be the same for calibration sources as for samples.

2.2.2.4 The sources used for the determination of self-absorption and cross talk should be of similar isotope content to that of the analytical samples. Site specific instructions may be provided regarding isotope content of samples and required isotopes to be used. If no direction is provided Am-241 or Th-230 shall be used for alpha and Cs-137 or Sr-90/Y-90 for beta.

2.2.3 Instrument Backgrounds

2.2.3.1 Instrument backgrounds for both alpha and beta shall be determined at least weekly.

2.2.4 Self-Absorption and Cross-Talk Curves

2.2.4.1 Self-absorption curves are required for both alpha and beta counting.

2.2.4.2 A cross-talk curve shall be established for alpha to beta cross-talk versus residue weight.

2.2.4.3 Beta to alpha cross-talk is not significantly affected by planchet residue weight, and is generally constant over the applicable weight range. Therefore this cross-talk correction does not require residue weight consideration.

2.2.4.4 The data used to generate self-absorption and cross-talk curves shall consist of at least 7 points, well distributed throughout the mass range.

2.2.4.5 Each alpha and beta calibration standard shall be counted to an accumulation of 10,000 counts.

2.2.5 Check Source Requirements

2.2.5.1 The alpha and beta calibration of each detector used to count analytical samples or QC samples shall be checked daily. The only exception to this requirement is when performing analyses with extended count times. In this case, check source measurements may be performed between sample sets.

2.2.5.2 Following gas bottle changes, check sources and backgrounds shall beanalyzed before samples are counted.

2.2.5.3 Check source data shall be documented and retained.

Part 5

TOTAL URANIUM BY LASER INDUCED KINETIC PHOSPHORESCENCE

1. RADIOCHEMICAL ANALYTICAL METHODS

Site specific instruction may be provided for other technology for determination of total uranium.

1.1 SELECTION OF METHOD

1.1.1 **Sample Treatment** Water samples shall be at least evaporated to dryness and wet-ashed as described in ASTM Specification D 5174-91, *Trace Uranium by Pulsed-Laser Phosphorimetry* prior to KPA measurement.

1.1.2 **Sample and Sample plus Spike Measurement** For each sample, both the sample and sample plus spike shall be measured to demonstrate that there are no quenching interferences.

1.2 GLASSWARE AND WATER FOR LOW LEVEL URANIUM

1.2.1 For all low-level uranium analysis, prior to initial use, all new glassware with the exception of cuvettes used in KPA measurement, shall be soaked in hot 8 molar nitric acid for at least two hours and then in room temperature 8 molar nitric acid overnight.

1.2.2 ASTM Type II water shall be used to prepare but is not limited to standards, preparation of all reagents, and for final rinsing of glassware for items used in the determination of low level uranium.

1.3 PREPARATION REQUIREMENTS

3.1.1 The sample preparation must yield samples such that lifetimes shall fall in the range of 150 μs to 350 μs.

2.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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2.1 MINIMUM DETECTABLE CONCENTRATION (MDC) DETERMINATION:

The MDC is a function of the sample size, variability of the preparation blanks, and instrument background which is a combination of low-level signals produced by sources other than the analyte including: photomultiplier tube dark count (electronic noise), luminescence from the quartz cells and optics, impurities in reagents, and stray ambient light.

2.1.1 **MDC Calculation:**

- The following equation shall be used to calculate the MDC:

$$MDC = [(4.65*S_b)*DIL]/V$$

where:

S_b = standard deviation(1s) of the blank population

DIL = dilution factor (ratio of total sample taken for preparation/aliquot measured in the cuvette)

V = sample volume or weight (liters or grams)

Units are in µg/l or µg/g

- The method of implementation of blank populations for calculation of MDCs shall be described in detail in an SOP which is accepted by the Site prior to use as described in Part 1, General Requirements of this SOW, Section 4. Site specific requirements for implementation of the MDC calculation may be provided.

2.1.2 The sample size used for blanks shall be the typical sample size for samples analyzed for this SOW. If a given sample size differs from the sample size used in the blanks, this shall be addressed in the Case Narrative. It is not expected that sample size will differ from the sample size of the blanks.

2.1.3 The analyte MDC's as calculated above must be less than or equal to the RDL at the lowest dilution factor.

2.2 CONDITIONS REQUIRING REANALYSIS

2.2.1 **Sample and Analyte Specific Conditions** the following are conditions that require reanalysis for a particular sample beginning with KPA measurement. If succeeding KPA measurement does not provide an adequate result, another aliquot must be started with the sample preparation.

2.2.1.1 The lifetime of the phosphorescence is less than 150 us or greater than 350 us.

2.2.2.2 The linear regression coefficient of the decay plot is less than 0.96 for samples where the measured concentration is greater than the RDL. Every effort should be made to keep this greater than 0.98. It is expected that for most samples this will be greater than 0.99.

2.2.2.3 If the standard addition recovery is less than 90%, re-analysis is required:

$$\text{Standard Addition Recovery} = (\text{Conc. Spike} - \text{Conc. Sample}) / \frac{\text{Std.Conc.} \times \text{Std.Vol}}{\text{Sampl. Vol.} + \text{Std.Vol.}} \times 100$$

2.2.2.4 Analyte concentration is not in the range of the calibration curve used.

2.2.2.5 The Reference Ratio is less than 0.9 or greater than 1.1.

2.2.2.6 The continuing Calibration Check Standard is not within 10% of the known value.

2.3 INSTRUMENT CALIBRATION

The purpose of the instrument calibration is to ensure that the KPA was initially capable of producing quality results according to the specifications given in this module and that the calibration was maintained throughout the time period in which samples were analyzed.

2.3.1 The KPA shall be calibrated daily when in use.

2.3.2 At least three standards shall be used for each calibration range. The calibration range shall include the range of the samples to be measured.

2.3.3 The LCS shall be measured in the same calibration range as the samples in the batch. Observe the instrument manufacturer's recommendations for calibrating high and low

ranges. If the measurements are performed in more than one calibration range, then a separate LCS shall be prepared for each range.

2.4 INSTRUMENT PERFORMANCE CHECK (also referred to as the Calibration Check Standard)

The performance check using a standard prepared separately and a different concentration from the calibration standards, shall be performed upon completion of calibration and subsequently after every 10 samples are analyzed. The relative bias of the calibration check standard shall be in the range -0.10 to +0.10.

2.5 INSTRUMENT CALIBRATION ORDER:

The order of performing the Instrument Calibration shall be (1) Background (2) Calibration Curve (3) Calibration Check standard

2.6 CALIBRATION REQUIREMENTS:

2.6.1 The background shall be sufficiently low to permit attaining RDL's.

2.6.2 R^2 (linear regression coefficient) for the calibration curve shall be greater than or equal to .99.

2.6.3 The Calibration Check Standard shall be within 10% of the known value.

Part 6
Gamma Spectrometry

1. RADIOANALYTICAL METHOD SELECTION

1.1 SAMPLE COUNTING REQUIREMENTS: Procedures for sample analysis by gamma spectrometry shall incorporate and adhere to ANSI N42.14-1991, *Calibration and use of Germanium Spectrometers for the Measurement of Gamma Ray Emission Rate of Radionuclides, and*/or ANSI N42.12-1994, *Calibration and Usage of Thallium-Activated Sodium Iodide Detector Systems for Assay of Radionuclides.* References are for the customer determined current version. When references change, an implementation schedule will be determined.

1.1.1 Detector Type: The gamma detector system shall consist of any detector suitable for measuring the gamma isotopes of interest in the range of $\leq$0.06 to $\geq$2 MeV with regard to attaining RDLs, bias and precision requirements. Specific guidance/requirements may be provided on a case-by-case basis. Ge detectors of either intrinsic (pure) germanium or lithium drifted germanium are preferred; however for some specific requirements, another detector type, such as sodium iodide, may be more appropriate.

1.1.2 Counting Geometry: Detectors shall be calibrated for the specific geometry and matrix considerations used in the sample analysis.

1.1.3 Spectral Acquisition, Processing and QC Software: The Laboratory shall identify the software package(s) and versions used to analyze Site samples in the Case Narrative. If the Laboratory uses commercially provided software unmodified to process spectra and calculate gamma spectroscopy MDAs, documentation shall be provided upon request. If the Laboratory has modified the commercial software, or uses in-house developed software, a description of the software or modifications shall be provided. The description shall include the algorithms and equations used for peak detection and fitting, nuclide identification, interference correction, energy and efficiency determination, and result, uncertainty and MDA calculation.

1.1.4 Spectral Data Reference: Identification of the reference used for the half-life, abundance and peak energy of all nuclides shall be documented.

1.1.5 Spectral Background: The count time for the background shall be at least as long as the sample count time. Background spectra shall be collected at the frequency prescribed for Instrument Calibration. A background shall also be collected after any counting chamber changes have been made, i.e. cleaning, liner replacement, or instrument modification.

1.2 SAMPLE PREPARATION REQUIREMENTS

1.2.1 Sample preparation shall follow the General Requirements given in Part 1.

1.3 RESULT REPORTING REQUIREMENTS

1.3.1 Result reporting requirements shall follow the General Requirements given in Part 1.

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2. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

2.1 MDA DETERMINATION

2.1.1 The method for calculating MDA is given in Part 1, General Requirements.

2.2 CONDITIONS REQUIRING REANALYSIS

2.2.1 The conditions requiring reanalysis are given in Part 1, General Requirements.

2.3 QC SAMPLE COUNTING:

2.3.1 All QC samples shall be counted and the spectra processed in the same manner as the samples in the Analytical Batch.

2.4 QC SAMPLES- BATCH BLANK (PB)

2.4.1 In the absence of Site specific instructions, the following batch blank matrices shall be used:

PREPARATION BLANK MATRICES

Sample Type	Blank Matrix Specifications
Water	Distilled or deionized water acidified to pH ≤ 2, radon free
Soil	Empty counting container.
Filters	Physically and chemically identical filter media (supplied by the Site)
Misc. Solids	Empty counting container.

2.5 QC SAMPLES- LABORATORY CONTROL SAMPLE (LCS)

2.5.1 The LCS shall be traceable to the National Institute of Standards and Technology (NIST) or shall be a working reference material as described in *ASTM C 1128* and may used repeatedly for different analytical batches as long as it is appropriate for the matrix and geometry of the batch.

2.5.2 The analyte need not be the same as the sample analyte but shall fall in the approximate energy region of the spectrum as the analyte(s) i.e. low, mid-range, or high energy.

2.6 INSTRUMENT CALIBRATION

2.6.1 Efficiency Calibration Requirements

2.6.1.1 Each gamma spectrometry system used for Site samples shall be efficiency calibrated for the sample geometry and matrix with NIST traceable standards or prepared from NIST traceable sources.

2.6.1.1.2 Germanium Detectors: Refer to *ANSI N42.14* for guidance on isotope specific efficiency and efficiency as a function of energy calibrations.

2.6.1.1.3 Sodium Iodide Detectors: Refer to *ANSI N42.12*.

2.6.1.2 Current software that does not require a physical calibration standard to obtain efficiencies for various matrices and geometries may be used to count samples where a standard calibration source of known matrix and geometry cannot be specified. This type of calibration technique is preferred for matrices such as waste or debris. When such software is used, the laboratory shall supply detailed information and documentation regarding the selection of parameters used to specify the efficiency calibration and sample models. Each sample selected for analysis using this type of calibration shall have a unique set of model parameters associated with it. When such models are used, the closest model to the actual sample shall be selected. The model selected for each sample shall be presented in the Case Narrative and shall include a discussion of actual and predicted peak ratios for isotopes with multiple gamma energies present in the sample. The model shall be Site accepted prior to use.

2.6.2 Energy Calibration Requirements

2.6.2.1 Germanium Detectors: Refer to ANSI N42.14, paragraph 5.3.

2.6.2.2 Sodium Iodide Detectors: Refer to ANSI N42.12, paragraph 4.3.2.

2.6.2.3 The energy calibration for each gamma spectrometry system shall be checked each day of use. For systems using sample changers and/or long count times that run more than a day, the energy calibration shall be checked before each Analytical Batch.

2.7 PERFORMANCE TESTING

2.7.1 Germanium Detectors: Refer to ANSI N42.14, paragraph 7.

2.7.2 Sodium Iodide Detectors: Refer to ANSI N42.12, paragraph 4.3.5.

2.8 SPECTRUM ASSESSMENT: Each sample and QC sample spectrum shall be assessed for acceptability of key peak width and shape, and interference due to superimposed peaks or other sources. Any major contributor to the spectrum that is an unidentified peak shall be discussed in the Case Narrative.

GLOSSARY

ACTINIDE SERIES: The series of elements beginning with actinium, element number 89, and continuing through lawrencium, element number 103.

ALPHA DECAY: The spontaneous emission of an alpha particle during radioactive decay of a nucleus. An alpha particle is a strongly ionizing particle from the nucleus having a mass and charge equal to that of a helium nucleus (2 protons and 2 neutrons).

ANALYTE: The particular radionuclide to be determined in a sample of interest.

ASTM TYPE I WATER: Reagent water with a conductivity of less than 0.1 µmho/cm at 25° C and has been polished with a 0.45 µm membrane filter. For additional specifications, refer to ASTM D1193-77, "Standard Specification for Reagent Water."

ASTM TYPE II WATER: Deionized water with a conductivity of less than 1.0 µmho/cm at 25° C. For additional specifications, refer to ASTM D1193-77, "Standard Specification for Reagent Water."

BACKGROUND: Ambient signal response recorded by measurement instruments that is independent of radioactivity contributed by the radionuclides being measured in the sample.

BATCH: A batch of samples is 20 samples or less plus a batch blank, a laboratory control sample, a duplicate and a matrix spike as appropriate. A batch of samples is processed throughout the entire analytical process together. If equipment restrictions limit the number of samples in any particular step, the samples in the batch are processed continuously and consecutively until the entire batch is completed.

BATCH BLANK: The batch blank is a laboratory-generated sample prepared with absence of the analyte of interest. Batch blanks are batch quality indicators and are carried through the entire sample analysis procedure with the samples in the batch.

BETA DECAY: The emission of a beta particle during radioactive decay of a nucleus. A beta particle is a charged particle emitted from the nucleus, having a mass and charge equal in magnitude to that of an electron.

BIAS: The deviation of a single measured value of a random variable from a corresponding expected value, or a fixed mean deviation from the expected value that remains constant over replicated measurements within the statistical precision of the measurement. (Synonyms: deterministic error, fixed error, systematic error.)

CARRIERS: Carriers are typically nonradioactive (e.g., natural strontium, barium, yttrium) elements. They follow similar chemical reactions as the analyte during processing and are added to samples to determine the overall chemical yield for the analytical preparation steps. The yield of the carrier is typically determined gravimetrically.

COUNTING EFFICIENCY: The ratio of the net count rate of a radionuclide standard source to its corresponding known activity.

COUNTING EFFICIENCY FACTOR: The fraction of actual disintegrations in the sample, which are counted by the detector as a function of residue weight.

CURIES: The traditional unit used to express the activity (amount) of radioactive material. The SI unit for activity is the becquerel.

1 curie (Ci) = 2.22×10^{12} disintegration/minute

1 millicurie (mCi) = 2.22×10^{9} disintegration/minute

1 microcurie (µci) = 2.22×10^{6} disintegration/minute

1 picocurie (pCi) = 2.22 disintegration/minute

1 becquerel (Bq) = 1 disintegration/second

DAUGHTER: A nuclide formed by radioactive decay of a parent radionuclide.

DUPLICATE SAMPLE: A second aliquot of a sample that serves as a Batch QC Sample, demonstrating analytical method precision and sample homogeneity.

EFFICIENCY: A measure of the fraction of actual disintegrations in the sample, which are counted by a detector.

ENERGY CALIBRATION: The correlation of the multichannel analyzer (MCA) channel number to decay energy, obtained from the location of peaks from known radioactive standards.

FIELD BLANK: A sample prepared in the field by transferring ASTM Type II Water to a clean sample container. The field blank is used to indicate the presence of contamination due to sample collection and handling.

GAMMA RADIATION: Electromagnetic radiation of nuclear origin usually accompanying another form of radioactive decay.

HALF-LIFE ($T_{1/2}$): The time required for 50 percent of a radioactive isotope to decay.

IONIZING RADIATION: Any electromagnetic or particulate radiation capable of producing ions directly or indirectly in its passage through matter.

KEY PEAK: a spectral peak used for identification or quantification of an isotope.

LABORATORY CONTROL SAMPLE (LCS): The LCS is a laboratory generated sample prepared by adding known quantities of analyte(s) to an appropriate matrix which contains no analyte activity and is carried through the entire analysis procedure with the samples. The laboratory control sample is a quality indicator and provides information about the relative bias of the analysis. It is used to assess the overall process for any inherent biases or trends.

MATRIX SPIKE: A matrix spike is an aliquot of a sample to which known quantities of analyte(s) have been added. It is carried through the entire analytical procedure with the samples in order to evaluate the appropriateness of the method for the matrix by measuring recovery of the added analyte(s).

MINIMUM DETECTABLE ACTIVITY (MDA): The minimum detectable activity is the smallest amount (activity or mass) of an analyte in a sample that will be detected with a probability β of non-detection (Type II error) while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). For the purposes of this SOW, the α and β probabilities are both set at 0.05 unless otherwise specified. (Reference: *ANSI N13.30 and ANSI N42.23*)

MIXED WASTE: Waste containing both radioactive and hazardous components as defined by the Atomic Energy Act and the Resource Conservation Recovery Act respectively.

NIST-TRACEABLE STANDARD: A Standard Reference Material (SRM) purchased either directly from the National Institute of Standards and Technology (NIST) or the other approved vendors who provide the traceability certificate to the NIST.

NUCLIDE: An atomic species characterized by the constitution of its nucleus, specifically by the number of protons and neutrons.

RADIOACTIVE DECAY: The process by which a spontaneous change in nuclear state takes place. This process is accompanied by the emission of energy and subatomic particles.

RADIOACTIVE WASTE: Solid, liquid, or gaseous materials containing radionuclides regulated under the Atomic Energy Act of 1954 as amended, and of negligible economic value considering recovery costs.

RADIATION YIELD: The amount of radiation of the type being measured that is produced per each disintegration, which occurs. For gamma spectrometry, this is commonly called gamma abundance.

REGION OF INTEREST (ROI): In radiochemical analysis, the Multichannel Analyzer region defining the isotope of interest displayed in terms of energy or channels.

RELATIVE BIAS: The quotient of the bias divided by the expected value.

ROUNDING RULES: If the number following those to be retained is less than five, the number is dropped, and the retained numbers are kept unchanged. As an example, 11.443 is rounded off to 11.44. If the number following those to be retained is five and if there are no numbers other than zeros beyond the five, the number is dropped, and the last place number retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44 while 11.425 is rounded off to 11.42. If the number following those to be retained is greater than five, the number is dropped, and the last place number retained is increased by one. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all numbers are carried though the calculations. Then the final answer is rounded to the proper number of significant figures.

SAMPLE DATA PACKAGE NARRATIVE (also known as the Case Narrative): The section of the sample data package describing all problems or unusual circumstances encountered in the analytical processing of the sample. The narrative should include descriptions of matrix interferences, dilutions required, explanations of any Quality Control deficiencies, method modifications and all other information that might affect the validation of the data.

SCINTILLATOR: A transparent substance that emits visible or near-ultraviolet light when traversed by an ionizing particle.

SITE: The Site as referred to in this document is the particular DOE facility responsible for a specific contract.

SPIKE: In radiochemical analysis, an accurately measured amount of tracer quantitatively introduced or transferred into a sample aliquot.

STANDARD REFERENCE MATERIAL: Material characterized by the U. S. National Institute of Standards and Technology (NIST) for the activity of radionuclides and issued with a certificate that gives the results of the characterization.

TOTAL PROPAGATED UNCERTAINTY: An estimate or approximation of the error associated with a measured value by propagation of individual uncertainties.

TRACER: A radionuclide that chemically mimics and does not interfere with the target radioanalyte through the chemical preparation and instrument analysis.

TRACER CHEMICAL RECOVERY: The percent yield of the recovered tracer radioisotope after the sample/tracer aliquot has undergone preparation and instrument analysis.

TRACEABILITY: Demonstrated lineage of measurement process quality to the national physical standards.

UNSUPPORTED NUCLIDE: A daughter nuclide, which has been removed from the parent(s) in the decay chain in which it was produced.

WORKING REFERENCE MATERIAL: a reference material usually prepared by a single laboratory for its own use as a calibration standard, as a control standard, or for the qualification of a measurement method. For further definition and requirements see *STD.ASTM C1128.*